

UNITED STATES PATENT OFFICE.

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PROPELLENT BULK SMOKELESS POWDER.

1,390,740.

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No Drawing.

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To all whom it may concern:

Be it known that I, RICHARD GEORGE WOODBRIDGE, Jr., a citizen of the United States, and a resident of Wilmington, in the county of New Castle and State of Delaware, have invented certain new and useful Propellent Bulk Smokeless Powder, of which the following is a specification.

This invention relates to an improved propellent powder of the bulk smokeless powder type and to a process of making such powder; it comprises subjecting grains of bulk smokeless powder to a prolonged polishing treatment with a deterrent such as graphite.

The surface of bulk smokeless powders is rough compared with that of dense smokeless powders. One object of my invention is to smooth this rough surface to reduce the liability of premature ignition. Another object of the invention is to so incorporate in the powder grains a certain amount of graphite as to impart to the powder new and desirable ballistic properties.

The term "bulk smokeless powders" is commonly applied to those smokeless powders which are so made that the charge for a 12-gage shot shell occupies the same space in the shell as the standard charge of 3 drams of black powder. Bulk smokeless powders differ from dense smokeless powders in that the latter are completely gelatinized by the use of solvents while bulk powders are only partially gelatinized. Inasmuch as many modern dense powders are made up containing a mixture of nitrocellulose soluble in ether-alcohol with nitrocellulose insoluble in ether-alcohol, the term "dense powders" also includes those powders in which the soluble nitrocellulose only is completely gelatinized. Dense powders are invariably made by either pressing the colloided nitrocellulose composition through a die by means of a hydraulic press or by rolling the colloided nitrocellulose composition into sheets, with subsequent granulating into grains.

Bulk smokeless powders on the other hand are invariably made by a process of manufacture in which grains of nitrocellulose composition are subjected to a partial har-

dening treatment by means of a solvent mixture, or the nitrocellulose is formed into grains by being agitated in water with a suitable solvent. Bulk powders are, generally speaking, not made by any process which subjects the nitrocellulose composition to the action of pressure, as by pressing the colloided nitrocellulose composition through a die by means of hydraulic pressure or by rolling the colloided nitrocellulose composition into sheets by means of steel rolls. Therefore, bulk powders owe their bulkiness both to the process of manufacture by which they are made and also to the fact that the nitrocellulose of the composition is only partially colloided. Because of the above, the surface of the bulk powders is rough and the partially colloided fibers of the nitrocellulose can be readily seen under a microscope and even with the naked eye.

Dense smokeless powders are invariably subjected to a glazing treatment with graphite, the object of which is to render the powder less liable to become ignited by electrification. The glazing treatment increases the gravimetric density of the powder and the coating of the powder grains with graphite causes them to flow more readily and to pack together closer.

Bulk smokeless powders are not graphited because it is not necessary as the grains are not electrified and there is little or no danger of ignition by electrification. This is probably due to the composition of bulk smokeless powders which contain in general nitrocellulose, starch, paraffin oil, barium nitrate and potassium nitrate.

The object of my invention is not merely to graphite bulk powders but is to give bulk powder grains a prolonged polishing treatment with graphite, the object of which is to smooth and polish the rough surface of the powder grains caused by the partially colloided fibers of the nitrocellulose. This polishing treatment is very unlike any graphiting treatment which is applied to dense smokeless powders and consists in a polishing treatment for many hours with a much larger quantity of graphite than commonly used for graphiting purposes. This treat-

ment not only serves to smooth the rough surface of the bulk smokeless powder grains but a very considerable amount of graphite is retained by the powder grains because of their porous nature. This graphite tends to retard the ignition of the powder and acts as a moderating agent. The effect of giving the surface of the powder a polishing treatment with graphite and the graphite retained by the powder decidedly improves the ballistic properties of the powder, as evidenced by a substantial decrease in pressure. For example, a sample of bulk smokeless powder before receiving my polishing treatment with graphite gave in the 12-gage shotgun with $1\frac{1}{4}$ ounces of #7 shot a mean velocity of about 975 feet per second, with a mean pressure of 6.75 tons per square inch, with a $3\frac{1}{2}$ dram load of the powder which weighed 43.12 grains. After the polishing treatment made as hereinafter described, a $3\frac{1}{2}$ dram load of the powder then weighed 45.40 grains and in the 12-gage shotgun under identical conditions as above gave a mean velocity of 905 feet per second at 40 yards, with a mean pressure of 3.98 tons per square inch. Besides this very decided decrease in ballistic pressure which was obtained, the powder left in the bore of the gun about one-half the residue ordinarily left in the bore of the gun by powder of similar composition but not subjected to my polishing treatment with graphite, and also gave equal if not better patterns.

The well known manufacturing operations of the so-called Schultze process of manufacture of bulk powders may be illustrated by the following typical example:—

A mixture composed of about 80 parts of nitrocellulose containing some 35 per cent. of water, 2 parts of starch, 3 parts of paraffin oil, 12 parts of barium nitrate and 3 parts of potassium nitrate, with or without added diphenylamine as a stabilizer, and with or without dye to color the powder, is prepared by mixing the several ingredients under edge runners, similar to those used in the milling of black powder. Water is added from time to time in order to keep the mixture moist. The resulting material is then passed through a sieve with circular meshes, the size of the openings of which are slightly larger than the size of the finished grain desired. This sieving breaks up the material into grains. These grains are dried and subjected to a partial hardening treatment by means of a mixture of denatured alcohol containing from 3 to 8% or more of acetone. The powder sprinkled with the solvent is sometimes stored in closed containers so that the solvent can effect a further colloiding of the nitrocellulose. The excess solvent is removed from the grains by drying at a suitable temperature, usually not exceeding 60° C., and the powder is conditioned by ex-

posure to the atmosphere or by the addition of moisture to give the powder grains the desired moisture content.

My new process may be illustrated by the following example:—

After the powder grains have been subjected to the partial hardening treatment described above with a mixture of denatured alcohol and acetone, and after the excess solvent has been removed from the powder grain by suitable drying of the powder, I then subject the powder grains to a prolonged polishing treatment with graphite. For example, a suitable quantity of the hardened powder grains are subjected to a polishing treatment by continuously agitating them in a suitable rotating drum or barrel, which may or may not be provided with ribs, for a period preferably not exceeding 24 hours although further treatment is not injurious, with from 1 to 10 lbs. of unctuous graphite for every 100 lbs. of powder grains. After the polishing treatment is completed the excess graphite is sieved from the powder, but owing to the porous nature of the bulk powder grains it will be found that from 50% to 75% of the graphite used for polishing will have adhered to or penetrated the interior of the powder grain. After sieving the powder to remove excess graphite, the powder is conditioned and is then ready for ballistic tests.

The amount of graphite which becomes united with the powder grains depends largely upon the duration of the polishing treatment, as well as upon the condition of the powder and the proportion of graphite used. In the above example, using 1 to 10 lbs. of graphite for each 100 lbs. of powder, the polished powder will ordinarily contain from 0.5% to 7% of graphite. I prefer a powder containing from about 2 to 4% of graphite.

I claim:—

1. The process which comprises agitating a mixture of at least partially colloided bulk smokeless powder grains and graphite.

2. The process which comprises subjecting bulk smokeless powder grains to prolonged agitation with graphite until said grains have become polished and a substantial proportion of graphite has become incorporated therein.

3. The process which comprises subjecting bulk smokeless powder grains to prolonged agitation with graphite until from 50 to 75% of the graphite present has become united with said grains.

4. A propellant bulk smokeless powder at least partially colloided and containing more than 0.5% of an explosive deterrent.

5. A propellant bulk smokeless powder at least partially colloided and containing more than 0.5% of an explosive deterrent located mainly on the surface of said powder.

6. A propellant bulk smokeless powder containing from about 0.5% to 7% of graphite having polished surfaces and containing from about 2 to 4% of graphite.
7. A propellant bulk smokeless powder having polished surfaces and containing more than 2% of graphite.
- 5 8. A propellant bulk smokeless powder
9. A propellant bulk smokeless powder 10 having polished surfaces and containing more than 2% of graphite.

In testimony whereof I affix my signature.

RICHARD G. WOODBRIDGE, Jr.